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TECHNICAL REPORT

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THE ACTION OF WATER VAPOUR ON ION IRRADIATED ALKALI
SILICATE GLASSES

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S U M M A R Y

The surface of ion irradiated alkali silicate glasses is sensitised to chemical attack by atmospheric water vapour. After initial evolution of hydrogen, this attack leads to the slow conversion of certain regions on the glass surface to water soluble corrosion products.

The observations suggest that the regions involved in this conversion are particles of a second phase, distributed throughout the main matrix, and they point to the importance of phase separation in determining the stability of glasses under irradiation.

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1. INTRODUCTION

In a previous publication(ref.1) we have reported that the surface of materials, containing elements from the first or second column of the periodic table, can be chemically sensitised by irradiation with either positive ions, electrons or photons. Upon exposure to atmospheric moisture hydrogen is evolved at such surfaces in what we have termed a primary reaction sequence. Once this has occurred, the products formed can in certain cases initiate a secondary corrosion reaction, the progress of which can be sustained even in the absence of any further irradiation.

As was shown earlier, the role of the irradiation in this sequence of events is the dual one of firstly breaking the bonds of the cation M^+ , which is either an alkali metal or an alkaline earth, and secondly that of creating suitable catalytic surface sites at which the reactions can take place.

Thus if M^+ is associated with an anion X^- , we represent the dissociation which occurs during irradiation by



The cation is in this manner removed from its normal position in the solid structure, and is free to move to an appropriate catalytic site, where electron transfer can occur, neutralising the ion, so that following exposure to atmospheric moisture hydrogen is evolved in the reaction



Likewise it is possible for the anion in equation (1) to react with another water molecule according to



This step completes the primary reaction sequence, at the end of which a hydroxyl radical remains available. The presence of this is a prerequisite for the secondary corrosion reaction. This reaction does not require the incidence of any further radiation, and can be initiated at suitable surface sites, to which the OH^- radicals have to migrate, and where they can break the M-X bond, so that



following which the newly formed X^- reacts with a further water molecule as in equation (3); again an OH^- is produced, and in this manner the secondary reaction cycle can in principle be continued indefinitely.

Our earlier work(ref.1) was primarily concerned with irradiation effects produced by positive ion bombardment in ionic crystalline substances such as NaCl, $CaWO_4$ and KH_2PO_4 . Only brief mention was made of similar reactions in alkali silicate glasses, and it is therefore the purpose of this article to report in more detail on irradiation effects in these materials. In doing so, we shall initially base our discussion on the random network model(ref.2,3), although it will become apparent later, that this model in its simple, unmodified form, cannot entirely account for our observations.

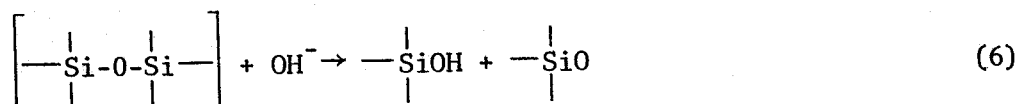
As is well known, the random network model can be discussed in terms of two substructures. The first of these (figure 1(a)) represents the continuous network, in which each "bridging" oxygen atom is linked by covalent bonds to two

silicon atoms. In addition there are terminal structures (figure 1(b)), through which the alkali ions are associated with the network. Here the "non-bridging" oxygen atoms are each linked to one silicon atom only, and their negative charge serves to bond the adjacent alkali ion. This ionic bonding is however relatively weak; removal of the alkali ion by incident radiation is readily accomplished, and if in addition the irradiation has also created suitable surface sites for electron transfer, reaction steps (1) and (2) can take place with the evolution of hydrogen and the formation of a hydroxyl radical.

With regard to the remainder of the reaction sequence, we propose that after removal of the positive ion the now truncated terminal structure is available for electrophilic attack by a water molecule(ref.4) described by



This step corresponds to reaction (3) above. The resulting OH^- radical may in turn initiate the corrosion reaction by breaking a Si-O bond in the continuous network according to



which is equivalent to equation (4) above. The second term on the right hand side of equation (6) represents a newly formed truncated terminal structure, and at such structures reaction (5) can be cyclically repeated with other water molecules.

The progressive breakdown of the continuous silica network represented by reactions (5) and (6) is part of a reaction scheme proposed earlier in a different context by Charles(ref.6). The process relies critically on the breaking of the strong Si-O bond, which in the undistorted oxygen-silicon tetrahedron has a bond energy of the order of 2.2 electron volts(ref.6,7). Any reaction involving the breaking of a bond must have an activation energy at least equal to the bond energy. One would therefore expect that if the reaction were to proceed at room temperature, it would do so extremely slowly. A faster rate would however be possible in regions of the surface, where the bond length had been altered e.g. by local changes in composition, or where as a result of mechanical damage (surface scratches etc.) dangling bonds had been created. As will be more fully discussed below, the corrosion reaction in alkali silicate glasses is in fact only observed in certain favourable surface regions, and even there the reaction rate in some glass types is so slow that the small agglomerates of corrosion products which are generated, become detectable under the light microscope only after many months of exposure to atmospheric moisture.

2. EXPERIMENTAL PROCEDURE AND OBSERVATIONS

As already stated, the reaction sequences of Section 1 can be initiated by ion, as well as by electron or photon irradiation. For reasons of experimental convenience however, the present work is confined entirely to ion induced effects, and bombardment of the glass specimen was carried out as described earlier(ref.1), using argon ions of 2000 eV energy, focussed into a beam of about 5 mm diameter at the target. With a total beam current of 40 μA , surface charging was neutralised by flooding the specimen with very low energy thermionic electrons.

Bombardments were carried out for periods ranging from 15 min to 1½ hours, and the effects produced were found to be essentially independent of the bombarding time in this range. During the irradiation, material near the beam centre, where the ion density was highest, was sputtered off at the rate of about 1.3 $\text{\AA}/\text{s}$.

Whilst most of the ion current was concentrated in the focal spot, the remainder of the target was nevertheless subject to very low intensity bombardment and, as in the earlier work, this low level irradiation caused a carbonaceous contamination film to be formed in the usual manner(ref.8) from hydrocarbon molecules present in the residual atmosphere of the vacuum system. Gas evolved in the primary reaction was contained under this film, where it formed into bubbles with diameters typically ranging from less than $1\text{ }\mu\text{m}$ to some tens of micrometers. It is on the basis of this direct evidence of a gaseous product, that we postulate a reaction represented by equation (2), in which the alkali takes part as a neutral atom rather than in its initial ionic state.

In comparison with ionic crystalline materials, where hydrogen evolution is practically instantaneous(ref.1), the primary reaction in glasses is very slow, and bubbles become noticeable after times which, depending on the glass type in question, may range from a few hours to several months. No detailed comparative study of the reaction rates has been made, but the general trend appears to be for more rapid reactions in glasses with higher alkali content, whilst no gas evolution at all has been detected in alkali free fused silica.

Very fast and vigorous response is obtained in commercial soft alkali lime glasses. Because of this, many of the observations reported here were made on glasses of this type, in particular on a glass readily available in the form of microscope slides. The composition of this latter material, of Chinese manufacture, is not known in detail, and it will be referred to in the following as type A.

In all glasses examined the distribution of gas bubbles across the surface has been found to be far less uniform than had previously been observed on crystalline materials(ref.1). Examples of the degree of non-uniformity encountered, both with regard to surface number density and to diameter of the bubbles, are given in the photographs of figure 2. These show two typical regions on the same target which, although only a distance of the order of 1 mm apart, exhibit widely differing amounts of gas evolution. Of particular interest in figure 2(a) are two features, marked by arrows, where rows of gas bubbles appear to be accurately aligned in straight lines. These lines are presumably associated with some kind of surface fault, which however could not be resolved under the light microscope. A similar association between the location of catalytic sites for the primary reaction and directly demonstrable surface damage has previously been established in ionic crystals(ref.1).

The total amount of gas generated, as well as its rate of evolution can be significantly increased by raising the target temperature. This is effective even after the ion bombardment has ceased, as demonstrated by the following experiment. A target of type A glass was bombarded in the usual manner at room temperature, and after exposure to the atmosphere for a number of hours it developed gas bubbles comparable in size and number density to those shown in figure 2. The sample was then baked for about $1\frac{1}{2}$ hours at approximately 180°C in a glass tube, evacuated by a rotary pump, and with the partial pressure of water vapour being kept low by a liquid nitrogen trap. At the end of the baking period the sample was microscopically examined through the glass envelope without breaking the vacuum, and the pattern of gas bubbles appeared to have undergone only little change. The liquid nitrogen was then removed from the trap, releasing the condensed moisture. With the rotary pump still operating, the target was left exposed to the moist residual atmosphere for 1 hour, during which period it cooled down to room temperature. At this stage the photographs of figure 3 were taken. To allow easier assessment of the effect of temperature increase, the target areas shown in figures 3(a) and 3(b) were selected so as to have roughly the same number density of bubbles as those of figures 2(a) and 2(b), corresponding to the room temperature case.

Comparison of the two sets of figures shows that even the moderate heating applied in our experiment caused a substantial and fairly rapid evolution of further gas, as judged from the much larger bubble diameter, which was attained in the relatively short time of only 1 hour. As remarked above,

no additional ion bombardment was required for this to occur. There is little doubt that in the first instance, and at room temperature, the primary reaction begins when the bombardment has dislodged cations in the manner we have described, but it would appear that once the irradiation has, in its second role, created catalytic sites for electron transfer, the reaction can be sustained at elevated temperature by alkali ions, which migrate to these sites after having been thermally displaced from their normal location. That a supply of mobile ions can be maintained by this mechanism is clear from the recent work of Vasile et al. (ref.9,10), who have demonstrated the high thermal mobility of Na^+ and K^+ ions in silicate glass structures, and who in particular have found that very substantial movement of Na^+ takes place during a $1\frac{1}{2}$ hour baking period at only 90°C (ref.10).

The dark zone on the left of figure 3(a) is a portion of the more intensely bombarded target area. Here the increased sputtering rate prevents the formation of a contaminant film (ref.8) under which the evolving gas can be collected. In this region therefore the generation of a gaseous reaction product is not easily observed, and the changes induced on the surface by irradiation are apparent only after a considerable period of time, when the corrosion products of the secondary reaction have accumulated to a sufficient extent.

For most vitreous materials examined, other than soft alkali lime glasses, this period was at least several months, and it was this rather long delay which led to our earlier incorrect conclusion that most glasses do not exhibit the effect at all. However, as an example to the contrary, figure 4 shows corrosion sites on Corning type 7059 glass, as they appear after 14 months exposure to the atmosphere. Under the light microscope these sites are visible only with difficulty, even at high magnification and with carefully optimised illumination. Their small size, estimated to be a few hundred nm, the long lapse of time before they become visible, and their patchy distribution on the surface, which permits them to be easily overlooked even when they are fully developed, have no doubt all contributed to the fact that the corrosion centres have not been reported before.

In contrast to this, on soft glasses, such as type A, the corrosion centres are not only more numerous and larger by an order of magnitude, but they can also be seen after a much shorter time. Thus figure 5 shows the appearance of an irradiated type A glass surface approximately one day after exposure to atmospheric moisture. On this sample the corrosion spots have "diameters" of about $1\text{ }\mu\text{m}$, and their number density is roughly $5 \times 10^6/\text{cm}^2$, but, as will be described further below, this number varies somewhat in different parts of the specimen surface.

It is possible to remove the spots by rinsing the sample for 2 min in distilled water; clearly therefore the corrosion products must be readily water soluble. If, instead of waiting for the corrosion centres to become visible, the rinsing is carried out within a few minutes of exposure to the atmosphere, the secondary corrosion reaction is entirely suppressed, and no spots are subsequently formed.

Examples of an interesting feature, occasionally found on irradiated glass surfaces with a fairly dense population of corrosion sites, can be seen on figure 5 in the form of several circular "lacunae" of roughly $15\text{ }\mu\text{m}$ diameter. Each of these lacunae is almost devoid of corrosion spots except for one particularly large accumulation at its centre, although closer examination of the circular area reveals that other reaction sites, although very small, are nevertheless present, and that within the lacuna their number density is in fact practically the same as elsewhere.

We believe that the phenomenon is due to the fact that, for reasons not yet understood, the central site is more effective than the neighbouring ones in "capturing" reactants as they diffuse across the surface. In consequence this corrosion spot will grow faster, and will accumulate material at the expense of sites in its vicinity.

Similarly to figure 2(a) the photograph of figure 5 also shows a practically linear arrangement of corrosion spots along a fault line. However, even in the absence of any recognisable macroscopic non-uniformity in the surface the corrosion spots are by no means randomly distributed, but are arranged in an intricate pattern of curve segments, with each segment having a length of the order of some tens of micrometres. As one moves over the irradiated surface, this pattern undergoes changes, examples of which are shown in the series of photographs of figure 6 which depicts corrosion spot patterns in a number of adjacent areas on a type A glass specimen.

On the left side of figure 6(a) one notices the weakly irradiated target region covered by a contamination film, with its boundary near the centre of the photograph. The patchy nature of the distribution of centres of gas evolution was such that on the area shown no bubbles are in evidence, but corrosion spots are noticed on the more intensely bombarded right hand side of the picture, and on the adjoining photographs 6(b) to 6(e). The size of these spots, their number density as well as their local distribution pattern are seen to vary. On the other hand measurements of ion current density rule out any possibility of these variations being due to non-uniformity in the bombardment intensity, so that the observed changes must reflect corresponding changes in the surface properties.

In particular, from variation in the number density of spots we can infer changes in the number of surface elements at which the corrosion reaction can proceed. This variation in site density is also shown in the histogram of figure 7, obtained by direct counting on the photographs which for this purpose were subdivided into vertical strips of 30 μm width. The limited spatial resolution introduced by this procedure tends to make the density changes appear somewhat less abrupt than is actually the case, a fact particularly noticeable from an inspection of figure 6(b), where very substantial variations in number density are found to occur over a distance of a few micrometres only.

It is also apparent from the figures that a relation exists between the size of the corrosion centres and their number density, with smaller spots being formed in regions of higher density, but whilst the photographs of figure 6 permit a qualitative comparison of the relative sizes of the spots, some caution is needed in any inference regarding their absolute magnitude and shape. This is so not only because of the limited resolution of the light microscope, but also as a consequence of the fact that after some time atmospheric moisture tends to accumulate at the corrosion centres, causing their apparent shapes to be distorted and enlarged, until ultimately they assume the appearance of small droplets. This is undoubtedly the phenomenon previously described by Primak(ref.11).

As an unfortunate consequence of the hygroscopic nature of the corrosion products it is usually rather difficult to examine the more detailed structure of the centres by means of the scanning electron microscope. Local heating by the electron beam will as a rule cause copious evolution of water vapour, and this in turn leads to bubbling and severe distortion of the conducting film, which is applied to the specimen to prevent surface charging. Under such circumstances details of surface topography cannot be observed, but in a few instances, with suitable atmospheric conditions, it has been possible to obtain micrographs of the centres; examples of which are shown in figure 8.

It is seen there that the corrosion sites are elongated structures of approximately 1 μm length. In some cases their shape is simply that of a "rod" with a diameter in the vicinity of 0.1 μm , and in many instances where the structure is more complex, it can to a fair degree of approximation be regarded as made up of a number of such rods. These vary somewhat in length and are inclined to each other at angles which frequently are small enough to allow a cardinal direction to be assigned to the "axis" of even a fairly complicated structure. In this sense it is possible to speak of a preferred orientation of the corrosion sites, and detailed examination of the micrograph reveals that with very few exceptions the structures are aligned along a small number of directions, all of

which point from the upper left to the lower right of the figure. As examples, two of the most prominent amongst these preferred directions may be defined by the axes of the two simple rod shaped structures marked by arrows.

Preferred orientation from upper left to lower right, as we have just described, is however only maintained over a small area of the target, and it certainly does not hold throughout the larger field of view in figure 8(b), taken at a lower magnification. Particularly if this figure is viewed at a grazing angle, it is possible to discern the curve segments along which the corrosion centres are arranged (c.f. figure 5), and it is seen not only that the preferred direction is different in various regions of the surface, but also that in any particular area this direction bears no obvious relation to characteristics of the curve segments, such as for example the direction of their tangent.

3. DISCUSSION OF RESULTS

In summary, the experimental observations of the previous Section show that the development of corrosion centres is associated with spatial ordering on three levels. Firstly the shape of the individual rod-like components of each corrosion centre indicates an essentially linear propagation of the corrosion reaction over distances of the order of $1\text{ }\mu\text{m}$. Secondly preferential orientation of the centres prevails over distances of roughly $20\text{ }\mu\text{m}$, and a third level of order is imposed by the corrosion sites being arranged on curve segments which extend over many tens of micrometres. Long range order on this scale is quite incompatible not only with the classical random network theory of the vitreous state (ref.2,3), but also with the crystallite model (ref.12,13), which envisages structural order over distances of little more than $10\text{ }\text{\AA}$.

Structural heterogeneity which can locally define specific directions on the required scale may however be provided through the phenomenon of phase separation, which is now known to occur in a large range of glass forming systems (ref.14). In this process one or more amorphous minority phases are formed, usually as small spheroidal particles embedded throughout the likewise amorphous matrix of the majority phase. The work of Seward (ref.15) has shown that during mechanical working of the glass near its softening point the viscous flow of the main phase material past the spheroids can cause the latter to be deformed into rod shaped elongated structures whose aspect ratios may range from the order of unity to well in excess of 100. It is interesting to note that evidence of this deformation effect can also be inferred from the early observations of Prebus and Michener (ref.16), who appear to have been the first workers to notice phase separation in glass.

Whilst the number density and size of the minority phase particles is dependent on factors such as glass composition and heat treatment temperature (ref.17,18), it is evident, particularly from the photographs of phase separation provided by Seward (loc. cit.) and Oberlies (loc. cit.), that in glasses subjected during manufacture to some type of forming process, minority phase particles are likely to be present, which closely resemble in both shape and size the rod-like structures of figure 8(a). We therefore postulate that these rod structures do in fact correspond to minority phase particles, the shape of which is rendered visible, when the secondary reaction converts the minority phase material into corrosion products, but leaves the majority phase essentially intact. One may also conjecture that the more complex shapes in figure 8(a) have their origin in initially existing groups of closely spaced second phase spheroids, which have simultaneously undergone elongation. Provided the flow pattern of the major phase material is not too complicated, one would expect such a deformation to produce corrosion centres composed of groups of practically parallel rod elements, and figure 8(a) shows that this is so in many cases. There are however in a few instances rather large departures from parallelism, the reason for which may need further examination.

An explanation of the corrosion sites along these lines could account not only for the observed preferential orientation of the individual centres, but also for their arrangement along curve segments, as being both due to the manner in which the glass flows in the course of the forming operation. Moreover it would appear from figures 6 and 7 that for materials such as soft alkali lime glasses, where the corrosion reaction proceeds rapidly, triggering this reaction by ion irradiation can provide a convenient method for demonstrating the presence of phase separation, and for estimating the number density and size distribution of the minority phase particles.

As has already been briefly mentioned in Section 1, our earlier work(ref.1) has established that, apart from dislodging the cation, a second function of irradiation is to render catalytically active certain suitable surface sites, where the reactions we have described will be initiated. Occasionally this activation can occur in areas of incidental surface imperfections, such as may for instance be caused by scratches, and the effect of structural faults of this type has already been referred to in connection with figures 2(a) and 5. However the vast majority of active sites is not associated with macroscopic surface damage. Thus, in crystalline materials it was found earlier that most of the sites arise at crystallographic ledges, where apparently the presence of dangling bonds serves to promote the reaction(ref.1).

It seems likely that in amorphous substances, with which we are here concerned, the role of crystallographic ledges is taken over by the compositional discontinuities, which exist at the boundaries separating the minority phase particles from the matrix, and that, once initiated at a boundary, the reaction will spread throughout the particle.

In our experiments no effect is discernible in the matrix itself, so that if the reaction does proceed there at all, it must do so at an exceedingly slow rate. The obviously much faster rate in the second phase points to a significantly lower value of the relevant bond strength there (c.f. Section 1), but in the absence of any information regarding the composition and other properties of the two separated phases, a more detailed understanding of the manner in which the corrosion reaction propagates through the material must await further investigation.

4. CONCLUSION

The experiments have shown that the surface of alkali silicate glasses is sensitised by irradiation to chemical attack by atmospheric water vapour. This leads in the first instance to the evolution of hydrogen, and subsequently to the slow conversion of certain regions of the glass into water soluble corrosion products.

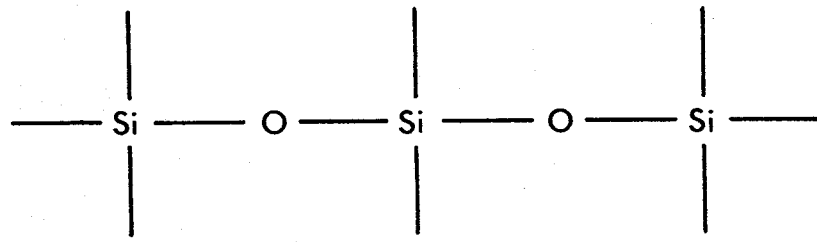
From the morphology of the regions in which this conversion occurs, it is concluded that they represent particles of a second phase, dispersed throughout the bulk of the glass. On the basis of this interpretation the observations point to the importance of phase separation in relation to the radiation hardness of glass, particularly when it is borne in mind that, although the experiments reported here relate to ion bombardment only, similar effects can also be induced by electron and photon irradiation.

5. ACKNOWLEDGEMENTS

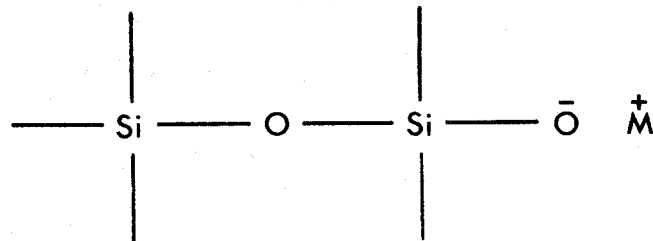
The authors wish to express their thanks to Mr L.J. Dunne for his interest and for stimulating discussions, and to Mr I.K. Varga for his contribution in the development of the experimental apparatus. Thanks are also due to Mr B.A. Johnson for his assistance in constructing the equipment and to Mr J.G.L. Terlet for preparing the electronmicrographs.

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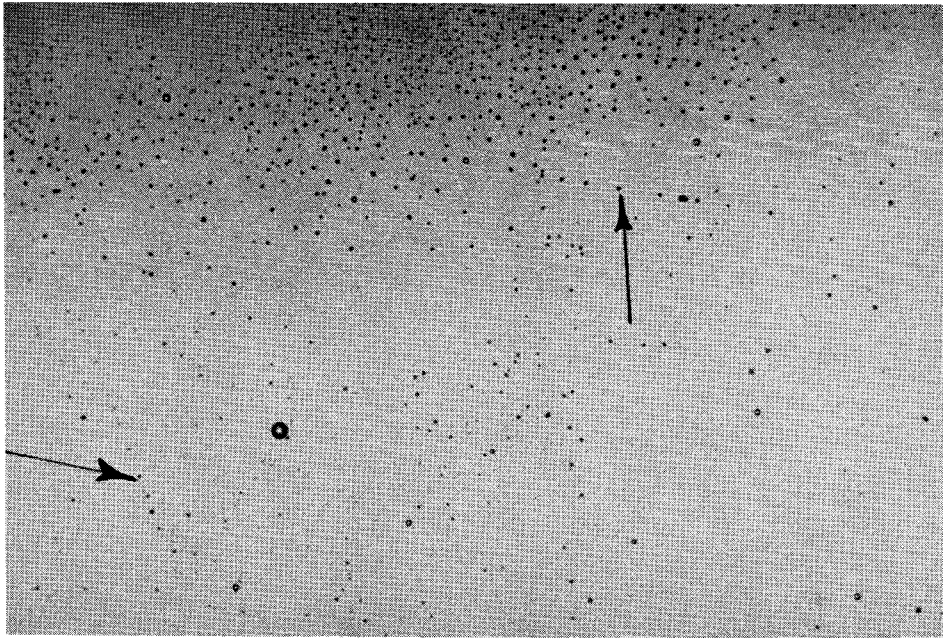


(a) Continuous Network

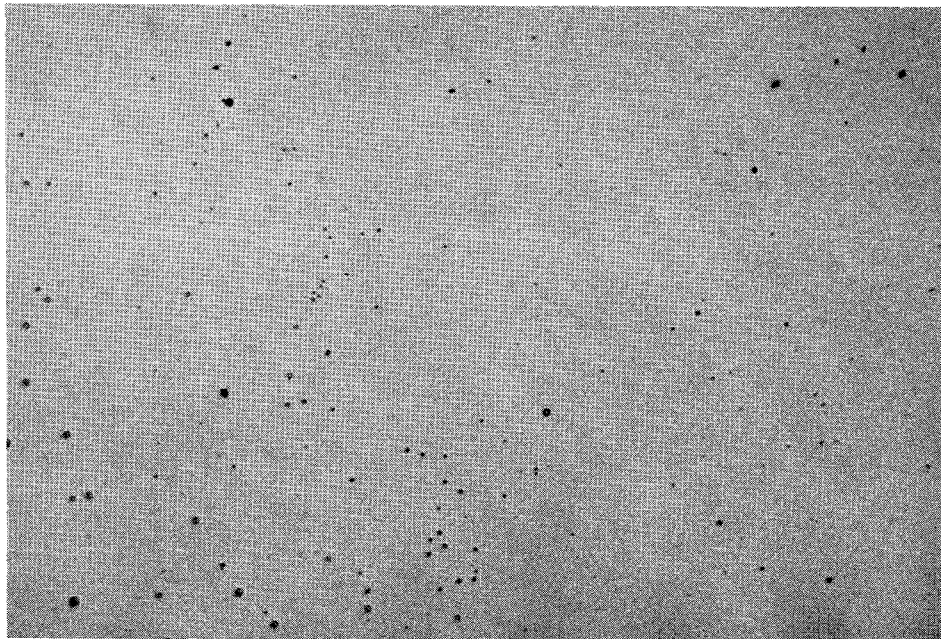


(b) Terminal Structure

Figure 1. Substructures in glass network



(a)

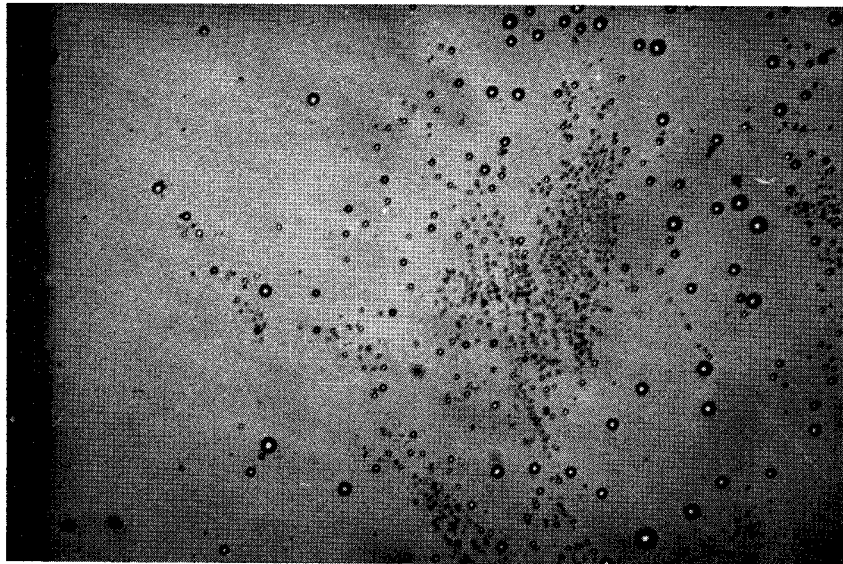


(b)

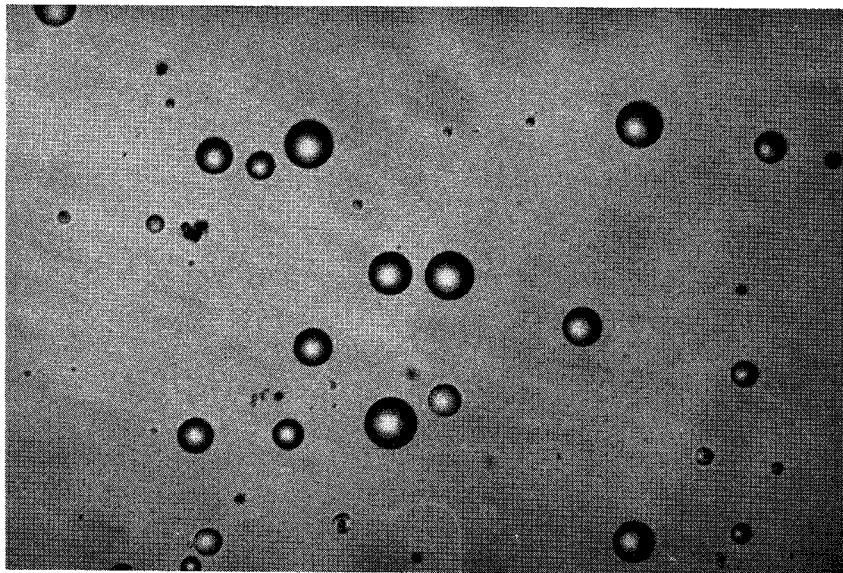
scale
0.2 mm

Arrows in figure 2(a) indicate linear arrays of bubbles

Figure 2. Typical target areas with gas bubbles (Type A Glass)



(a)



(b)



scale

0.2 mm

Figure 3. Typical target areas showing effect of heat treatment on development of gas bubbles (Type A Glass)

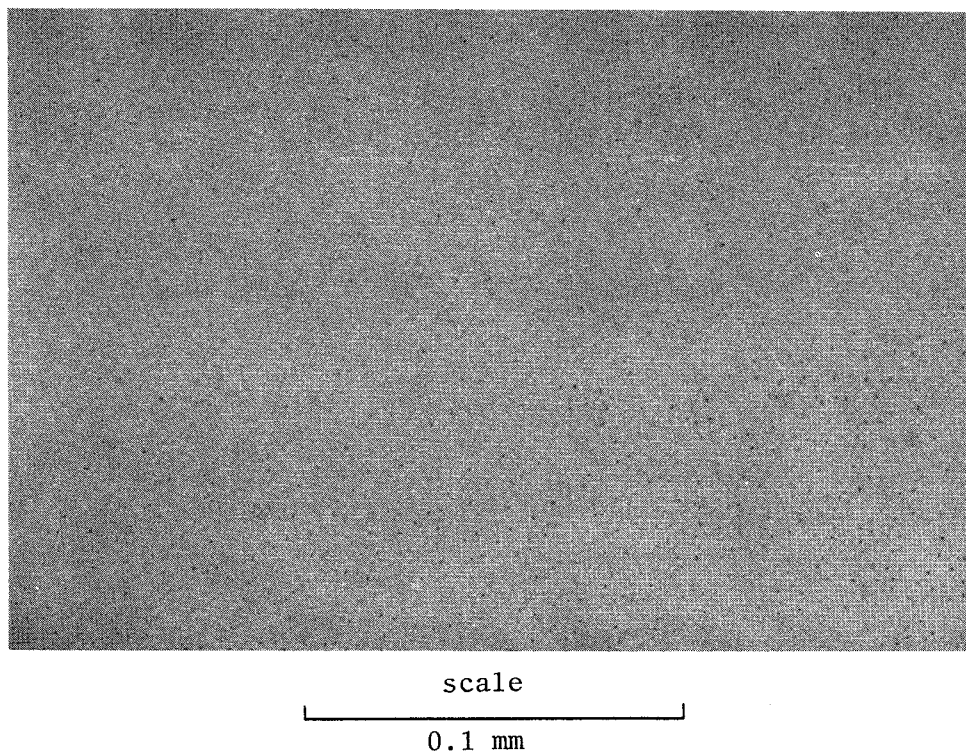


Figure 4. Corrosion centres on corning glass, Type 7059

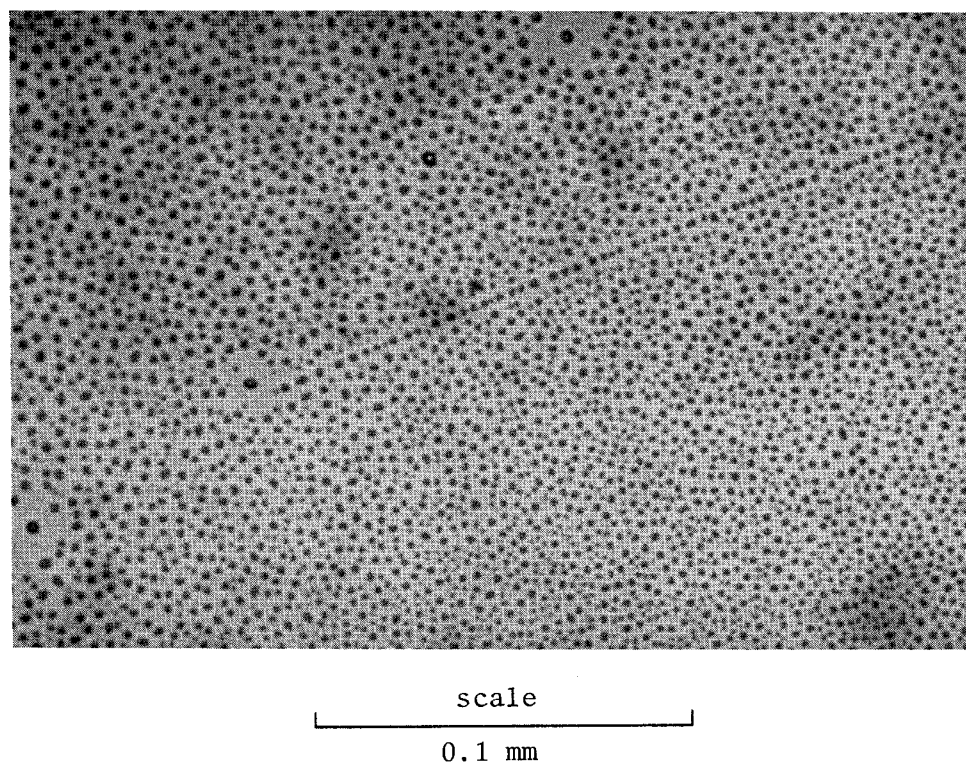
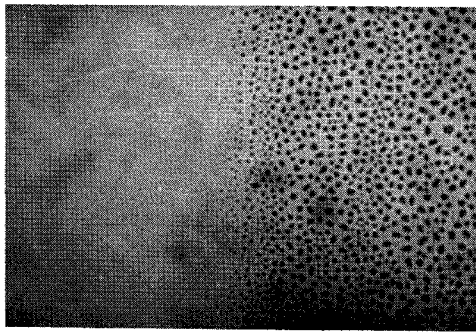
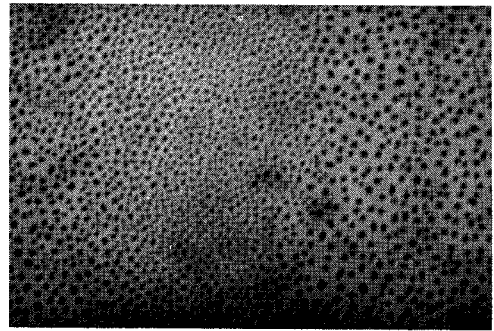


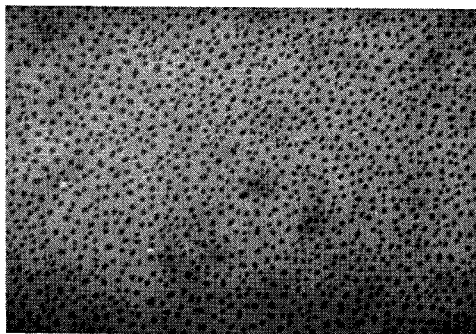
Figure 5. Typical type A glass target with corrosion centres



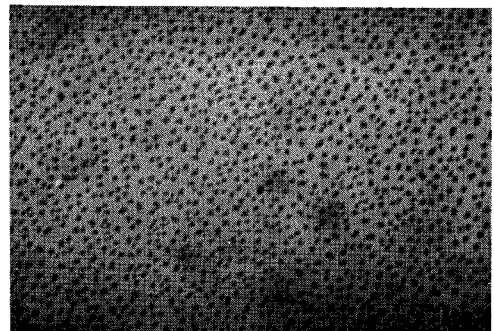
(a)



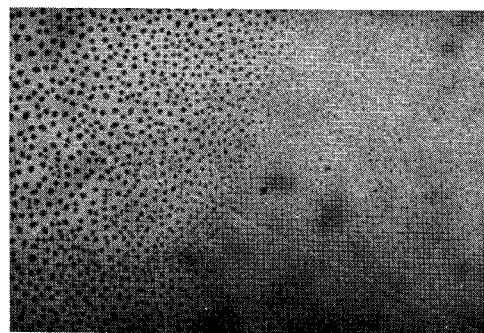
(b)



(c)



(d)



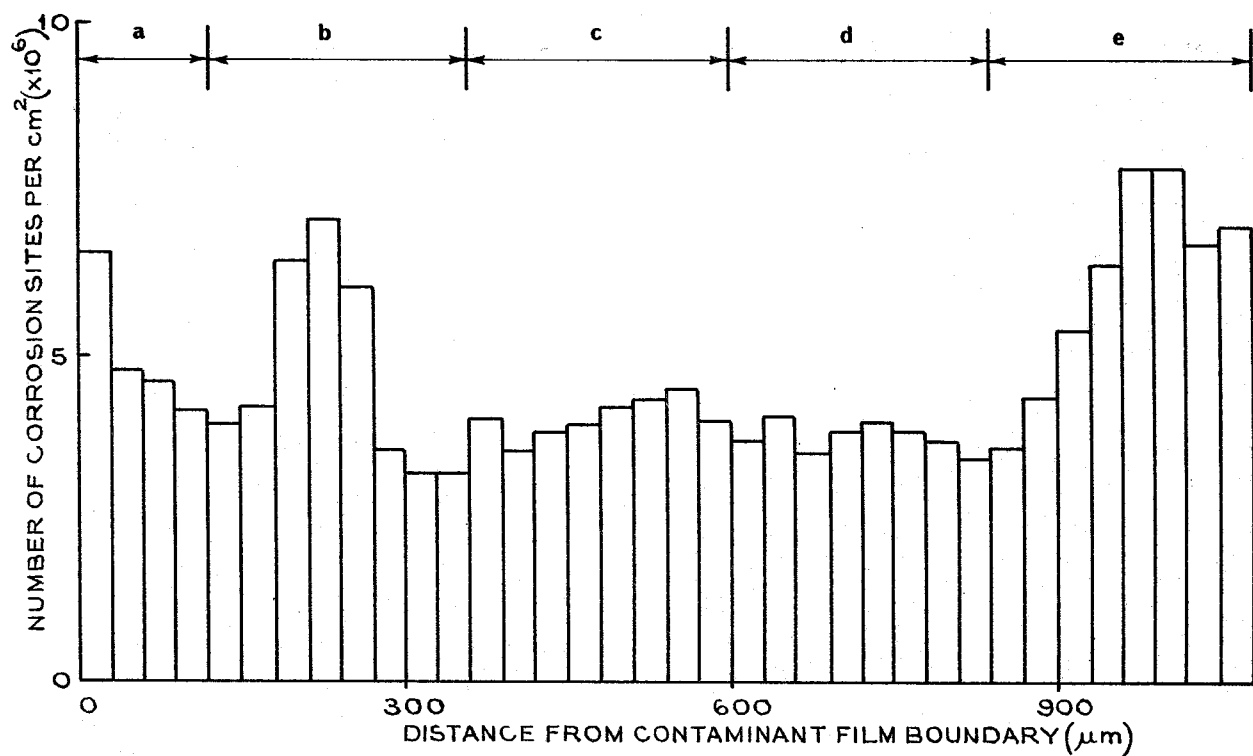
(e)



scale

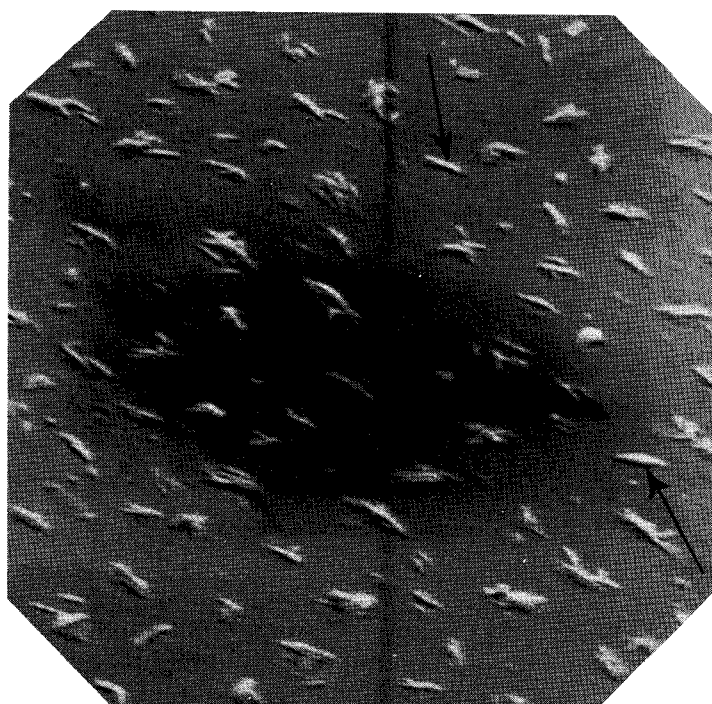
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Figure 6. Variation of corrosion site pattern across type A glass target



Letters a to e refer to corresponding photographs of figure 6.

Figure 7. Histogram of corrosion site density (Type A Glass)

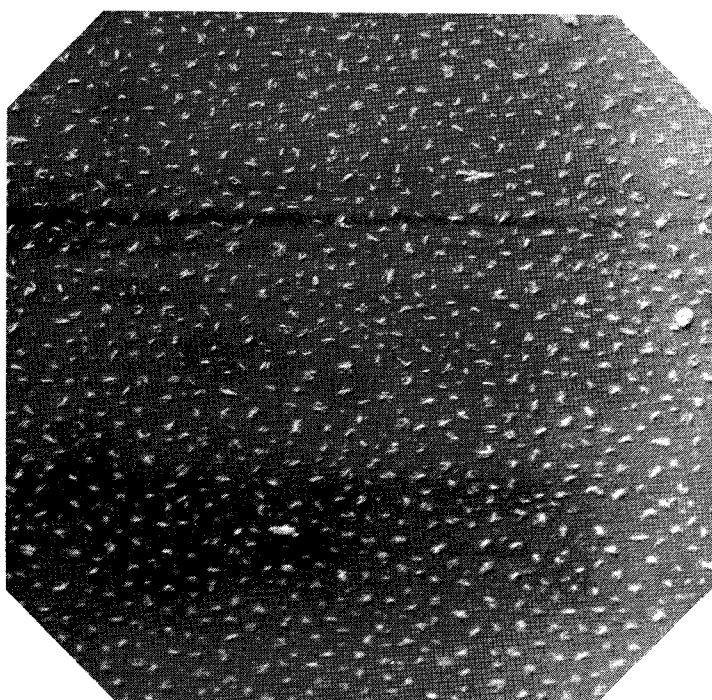


(a)

scale



10 μm



(b)

scale



50 μm

The rod shaped structures marked by arrows in figure 8(a) are aligned in two of the most prominent preferred orientations.

Figure 8. Detailed structure of corrosion centres (Type A Glass)

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The surface of ion irradiated alkali silicate glasses is sensitised to chemical attack by atmospheric water vapour. After initial evolution of hydrogen, this attack leads to the slow conversion of certain regions on the glass surface to water soluble corrosion products.

The observations suggest that the regions involved in this conversion are particles of a second phase, distributed throughout the main matrix, and they point to the importance of phase separation in determining the stability of glasses under irradiation.